U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
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Attn: Dr. N.G. Roman

The following is a quarterly progress report for the period January 1, 1966 to March 31, 1966 on NASA Contract No. R-73. This report was delayed slightly so that a technical report on the scattering of substrates and films in the far uv could be included.

a. Exploitation of the NBS synchrotron as a far ultraviolet light source

The 3-meter grazing-incidence scanning monochromator has been in operation on the synchrotron throughout this period. With this instrument we have now analyzed the profiles of many resonances in the photoionization continua of helium, neon, argon and xenon. We have also utilized this instrument to study the attenuation of aluminum at the $L_{II\ III}$ edge (~ 171 Å) and the transmission of an interesting filter for the 250-500 Å spectral region comprised of a Al-Mg-Al sandwich.

b. Investigation of thermal detectors as primary radiation standards for the vacuum ultraviolet.

A Sampson-type double ionization counter detector has been constructed and operated with the rare gases as an absolute radiation detector. This is now working quite satisfactorily. Construction of a facility to interchange a thermopile detector with the ionization detector in the same monochromatic beam is now completed. Thus we are now very close to the comparison of these two independently calibrated absolute detectors. Effort on this project has been stepped up to realize this meaningful comparison before the move to Gaithersburg.

c. Scattering measurements

The measurements of the radiation scattered by substrates and evaporated films in the far ultraviolet have been completed and a technical report on this work is included with this letter. The scattering at 1216 Å and 584 Å of fused silica and of various glasses with different degrees of polish were determined, and as well the growth of scattering with film thickness for both aluminum and gold. Of the substrates studied, highly polished silica was found to have the lowest scatter and gold was found to scatter considerably less than aluminum for the same film thickness. It was also found that the growth of scatter with film thickness was considerably less if the films were deposited on substrates having low scatter.

Sincerely,

R P Madden

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Encls.

cc: Miss W. Morgan (25)
L. Dunkelman

Scattering from Substrates and Evaporated Films in the Far Ultraviolet

by

R.G. Johnston, L.R. Canfield and R.P. Madden

ABSTRACT

Measurements of radiation scattered from mirror surfaces have been made at the wavelengths 1216 Å and 584 Å. The method utilized a relatively small area detector which was scanned in angle about the sample substrate, and a subsequent integration over the scattering angle. Results have been obtained from several glass and fused silica substrates with different degrees of surface polish, and from evaporated films of aluminum and gold as a function of film thickness. The results indicate that fused silica can be polished to a smoother surface than can be achieved with glass, that a smooth substrate surface is significantly advantageous in obtaining evaporated films having surfaces with relatively little scatter, and that gold films are considerably smoother than aluminum films of equal thickness.

Introduction

It is generally desirable to minimize the radiation scattered from optical surfaces. Scattered light reduces the effectiveness of gratings and mirrors, and the analysis of reflection data to determine the optical properties of thin film materials generally will yield improper results unless scattering is low.[1,2] Although much attention has been given to scattering in other wavelength regions (see, for example, Refs. 3 & 4), there is no available guide to the quality of optical surfaces required to meet these needs in the far ultraviolet.

This paper reports the measurement of the radiation reflectively scattered from various quality optical surfaces in the far ultraviolet. Further, the scattering from these surfaces has been measured before and after overcoating with various thicknesses of aluminum and gold. Thus these two materials can be compared, and the effect of film thickness on the degree of scattered radiation can be determined.

Apparatus

A dc gas discharge light source used with a near-normal incidence vacuum ultraviolet monochromator provided the incident radiation for these measurements. Attached to the exit arm of the monochromator was a vacuum system in which a sample holder and a Bendix M306 magnetic multiplier could be independently rototated about a common axis

intersecting the light beam from the monochromator. The sample holder could be withdrawn from the beam to permit the direct measurement of the beam.

The detector which was masked to an area of uniform response, was operated at a gain of about 10^4, and the output current was kept below 1×10^{-8} amp. For these low currents the detection system was found to be linear to a few percent over five orders of magnitude. The apparatus was located in a shielded room enabling the measurement of light intensities producing currents an order of magnitude below the multiplier dark current.

Procedure

The monochromator exit beam was stopped down to about f/50. Thus stopped, the area of the sample illuminated was on the order of 2.5 x 2.5mm. With the detector in the direct beam and the sample removed, the area illuminated at the detector was about 3.5 x 3.5mm. The active detector dimensions were larger than this by a factor of three in both directions, and subtended a solid angle at the sample of about 3.5×10^{-2} steradians.

The detector was first scanned through the direct beam, producing an angular intensity profile of the beam smeared by the detector function. This profile determined, within a normalization factor, the expected profile for a scan through the reflected

beam from a perfect specular reflector. The normalization was obtained by measuring the reflectance, R_m, of the sample at an incidence angle of 15 degrees. Since the samples were primarily specular reflectors, the error in measuring the specular reflectance with a detector larger than the reflected beam (due to including some of the scattered light) was small.

Having thus established the expected angular intensity profile of the reflected beam for a perfect specular reflector, the actual angular distribution of the reflected radiation was measured. The angular range covered was from 10 degrees off the specular reflection direction out to the largest scattering angle at which a significant signal could be recorded. The measured values were plotted together with the "perfect reflector" profile - an example is shown in Figure 1. Repeated measurements, with the sample rotated within the plane of its surface, assured that the scattering distribution was isotropic. The difference between these two profiles (Fig. 1) is then assumed to be the intensity of the scatter signal, $I_s(\theta)$, and the total scatter, expressed as a fraction of the incident intensity, is calculated from the equation:

$$S = \frac{2\pi r^2}{AI_o} \int_{10°}^{90°} I_s(\theta) \sin\theta d\theta$$

where A is the detector area, r is the radius of the detector movement, θ is the scattering angle and I_o is the incident beam intensity.

The use of this integration procedure assumes that the distribution of scattered radiation is symmetric about the direction of specular reflection. The fact that the quantity S obtained was found to be insensitive to the angle of incidence between 6° and 20° indicates that this assumption is at least a first order approximation. Notice that the integration extends only to within 10 degrees of the specular reflection direction. The fact that we are evaluating only the radiation which is reflectively scattered through an angle greater than this is important in the quantitative interpretation of the results.

A convenient evaluation of the lack of smoothness of the optical surface in question, which is not too dependent† on the reflectance of the surface, is the quantity:

$$f = \frac{S}{R_m + S}$$

where R_m is the measured value of the specular reflectance.

Here the denominator represents the total flux coming from the surface in question, and the ratio, f, is the fraction of that flux which is scattered more than 10° from the specular direction.

†The quantity to be defined, f, will in fact, vary somewhat with changes in the functional relationship between reflectance and incidence angle, and becomes difficult to interpret if interference effects are present.

Results

The quantity f was determined for highly-polished fused silica and for glasses with various degrees of polish. The measurements were made at the two wavelengths, 584 and 1216 Å, resulting in the data presented in Table I.

The highly-polished fused silica samples showed significantly less scatter than any of the glasses measured, even when the same polishing technique was used. However, the scattering from all samples is sufficiently low that if a correction were not applied, the error in measuring the specular reflectance would be no greater than about 1% of the value of the reflectance down to a wavelength of 584 Å. (This implies that all radiation scattered less than 10° from the specular direction is included in the specular reflectance.)

When such substrates are overcoated with evaporated films the value of f changes. The results of coating commercial plate glass (as sold) and the same type of glass after it had been given a high optical polish are shown in Fig. 2. The aluminum used was 99.99% pure and was evaporated at a deposition rate of about 150 Å per sec., maintaining a pressure during the evaporation of below 2×10^{-5} mm Hg. The measurements shown in Fig. 2 were taken at 1216 Å. The values of f for zero film thickness indicate the differences in the substrate surfaces. As the film thickness increases, the scattering increases, and the increase is more rapid in the case of the rougher substrate. Results using gold and using

aluminum at other wavelengths indicate this effect consistently. At 1216 Å, aluminum films of 700-900 Å are essentially opaque; however, if thicker coatings are required for special applications, a very smooth substrate would yield a significant advantage.

In Fig. 3 the scattering of gold films is indicated as a function of thickness at 1216 Å. The scattering of aluminum films when deposited onto similar substrates is shown for comparison. The substrates were measured before coating to assure that they had essentially the same value of f. Fig. 3 indicates that gold overcoating does not increase the scattering of the substrate until the thickness becomes quite large. The advantage of gold over aluminum in this respect is quite significant. This advantage would be expected to be even greater at the shorter wavelengths where the scattering from both materials would be larger. This fact is undoubtedly responsible, in part, for the noted high quality performance at short wavelengths of gratings ruled in gold.[5]

The wavelength dependence of scattering is indicated in Fig. 4 for gold films deposited onto plate glass substrates. The results obtained at 1216 Å (also shown in Fig. 3) are compared with those obtained at 584 Å. The scattering factor, f, is, of course, larger at 584 Å than at 1216 Å, and also, the increase with film thickness is more rapid at the shorter wavelength. At 3000 Å thickness the scattering from gold at 584 Å is rather significant.

The scatter in the points for the 584 Å measurements is greater than for the 1216 Å data due to a weaker and somewhat less stable source of 584 Å radiation. Considering the magnitude of the scatter from gold at 584 Å and the difference between gold and aluminum seen in Fig. 3 for 1216 Å, one would expect the scattering from thick aluminum films to be quite severe at 584 Å. In fact, f values on the order of 10% have been obtained for 3000 Å thick aluminum films. Data on aluminum at 584 Å have not been presented, however, due to the complication in interpretation caused by the transparency of aluminum at this wavelength.

Conclusions

The scattering ratio, f, represents the fraction of the total flux coming from the sample which is scattered through an angle from the specular direction of more than 10 degrees. Thus the quantitative interpretation of f, is as a lower limit to the total fraction of flux scattered. However, for many practical purposes the value of f should be of quantitative significance. A consideration of the relative values of f yields the following conclusions. First, fused silica can be polished to a significantly smoother surface than obtainable with glass. Second, for both aluminum and gold coatings, the roughness of the film surface increases more rapidly with increasing film thickness for rougher initial substrate surfaces. And third, gold films increase in roughness with increasing film thickness much less rapidly than aluminum films.

Table I

Sample	f (%)	
	1216 Å	584 Å
fused silica - pitch polish	0.07	0.19
window glass[a] - as sold	0.20	0.72
plate glass[b] - as sold	0.22 - 0.44	0.98 - 1.4
plate glass[b] - Cerox polish	0.23	0.86
plate glass[b] - cloth polish	0.26	1.2
plate glass[b] - pitch polish	0.17	0.42

a) LOF "Pennvernon"

b) PPG "water white"

Values of the scattering factor, f, for fused silica and several glasses with different degrees of polish at the two wavelengths, 584 Å and 1216 Å.

Figure Captions

Fig. 1 Angular distribution of flux reflectively scattered from a commercial plate glass substrate coated with 3000 Å of gold, at λ1216 Å. The observed angular profile is compared with that expected from a "perfect" reflector. The scattering angle, θ, is measured relative to the specular reflection direction. Angle of incidence, 10 degrees.

Fig. 2 Influence of substrate smoothness and film thickness on the scattering from evaporated aluminum films at λ1216 Å. Substrate A was commercial plate glass (as sold). Substrate B was the same glass given a high optical polish before deposition of the aluminum.

Fig. 3 Scattering of evaporated films of gold and aluminum as a function of film thickness at λ1216 Å. Both materials were deposited on commercial plate glass substrates.

Fig. 4 Scattering of evaporated gold films at λ584 Å and 1216 Å as a function of film thickness. Substrates were commercial plate glass.

References

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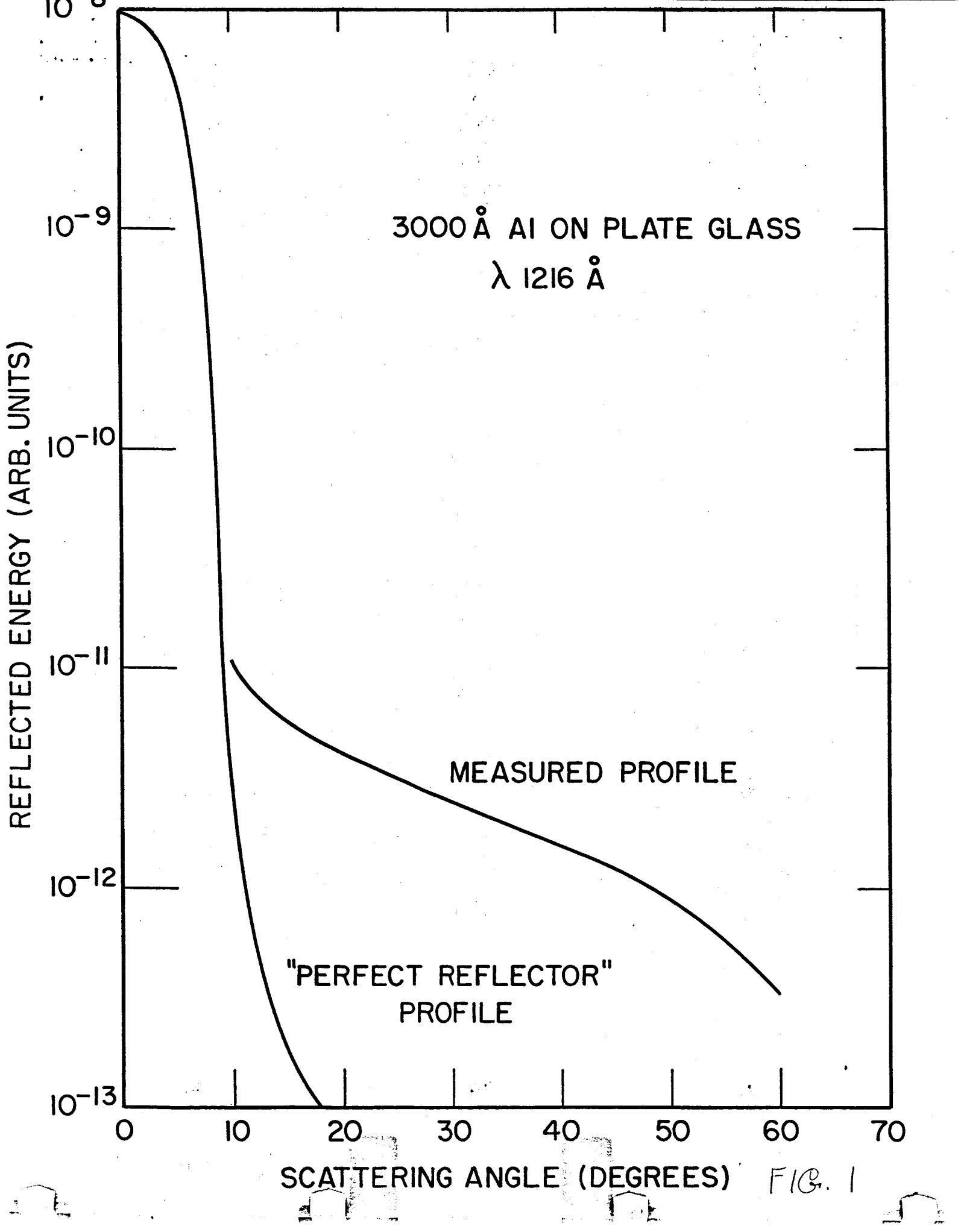
3000 Å Al ON PLATE GLASS
λ 1216 Å
MEASURED PROFILE
"PERFECT REFLECTOR"
PROFILE
REFLECTED ENERGY (ARB. UNITS)
10⁻⁹
10⁻¹⁰
10⁻¹¹
10⁻¹²
10⁻¹³
0
10
20
30
40
50
60
70
SCATTERING ANGLE (DEGREES)

FIG. 1

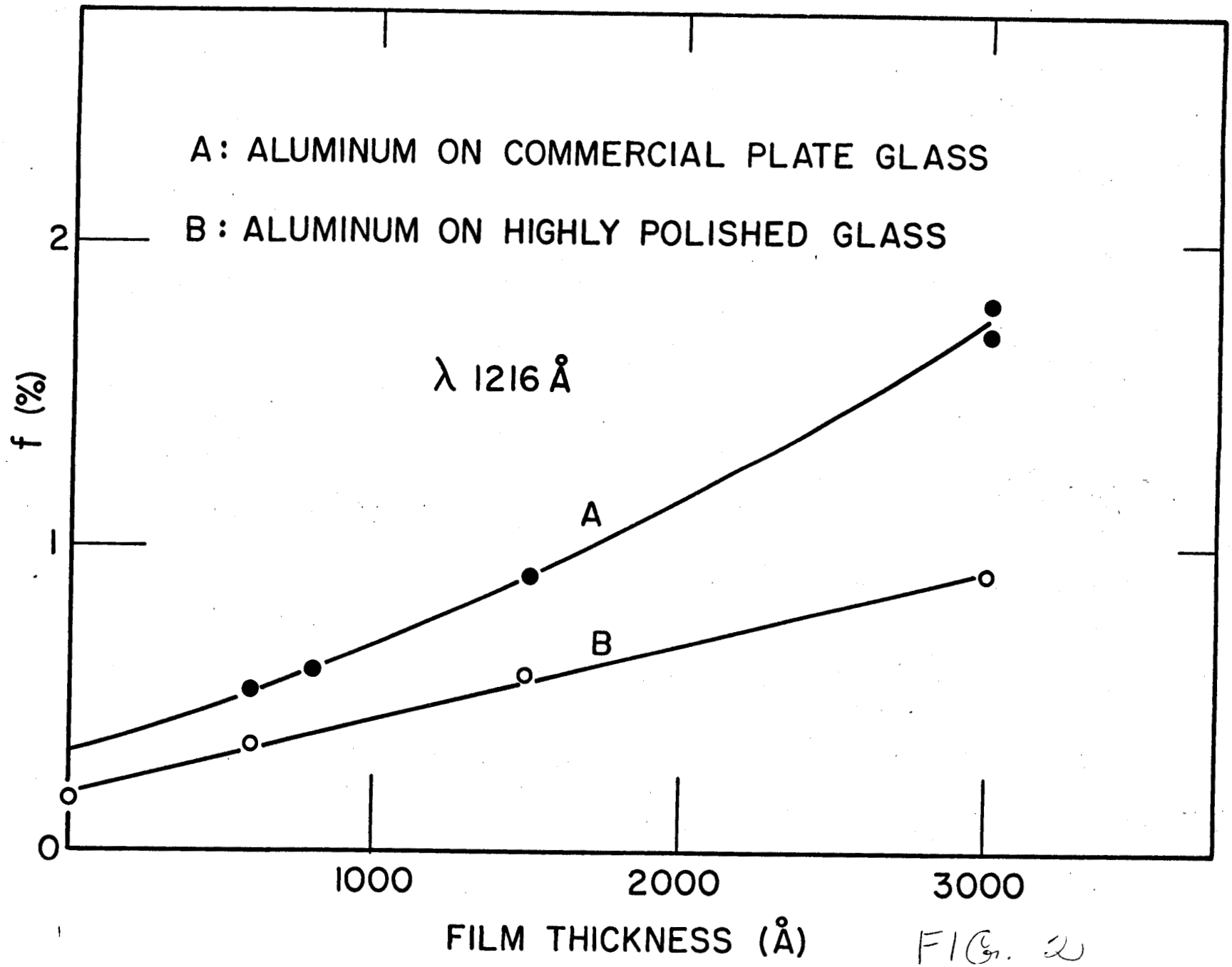
A : ALUMINUM ON COMMERCIAL PLATE GLASS
B : ALUMINUM ON HIGHLY POLISHED GLASS
λ 1216 Å
f (%)
2
1
0
A
B
1000
2000
3000
FILM THICKNESS (Å)

FIG. 2

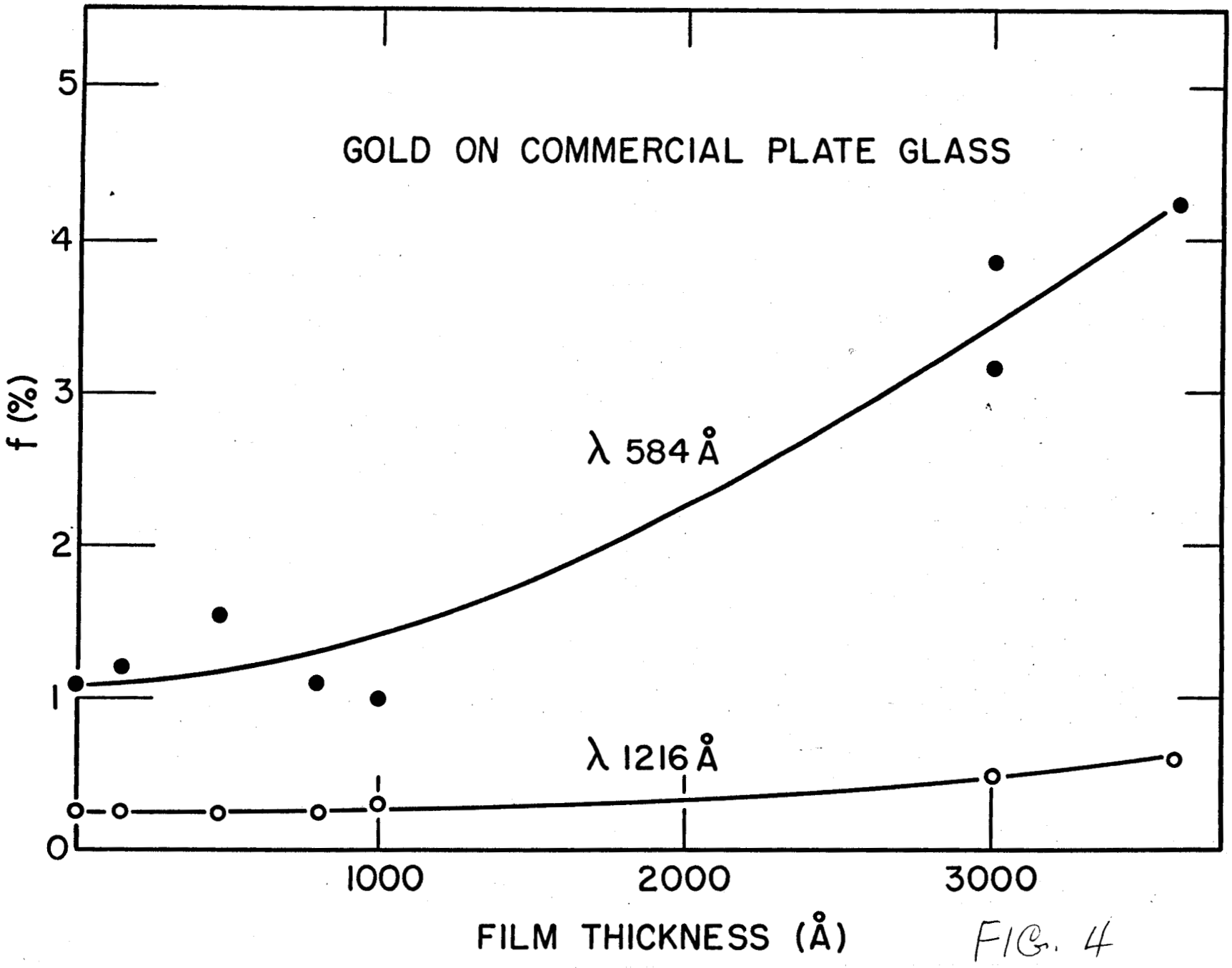
GOLD ON COMMERCIAL PLATE GLASS
λ 584 Å
λ 1216 Å
f (%)
0
1
2
3
4
5
1000
2000
3000
FILM THICKNESS (Å)

FIG. 4